Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# (2*E*)-2-[(3*E*)-4-Phenylbut-3-en-2-ylidene]-hydrazinecarboxamide

**S. Samshuddin,[a] Ray J. Butcher,[b] Sema Ozturk Yıldırım,[c] Mehmet Akkurt,[d]* B. Narayana[a] and H. S. Yathirajan[e]**

[a]Department of Studies in Chemistry, Mangalore University, Mangalagangotri, Mangalore 574 199, India, [b]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, [c]Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA, and, Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, [d]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, and [e]Department of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India
Correspondence e-mail: akkurt@erciyes.edu.tr

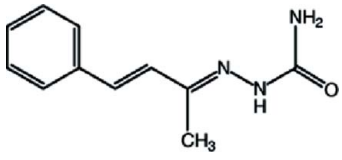
Received 5 December 2011; accepted 6 December 2011

Key indicators: single-crystal X-ray study; $T$ = 123 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.055; $wR$ factor = 0.176; data-to-parameter ratio = 21.0.

In the title compound, $C_{11}H_{13}N_3O$, the phenyl ring is disordered over two sites, with occupancy factors in a 0.520 (17):0.480 (17) ratio. The dihedral angle between the ring planes of the major and minor components of the disordered ring is 12.9 (2)°. In the crystal, molecules are linked by N—H···O hydrogen bonds, forming $R_2^2(8)$ ring motifs. C—H···O, C—H···N and C—H···$\pi$ interactions also occur.

## Related literature

For background to the biological activity of semicarbazones, see: Beraldo *et al.* (2002); Teixeira *et al.* (2003); Du *et al.* (2004); Kucukguzel *et al.* (2006); Beraldo & Gambino (2004). For related structures, see: Naik & Palenik (1974); Wang *et al.* (2004); Yathirajan *et al.* (2006); Sarojini *et al.* (2007).




## Experimental

### Crystal data

$C_{11}H_{13}N_3O$
$M_r$ = 203.24
Monoclinic, $C2/c$
$a$ = 15.1094 (8) Å
$b$ = 24.4445 (11) Å
$c$ = 7.0368 (4) Å
$\beta$ = 109.908 (6)°
$V$ = 2443.7 (2) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.07 mm$^{-1}$
$T$ = 123 K
0.40 × 0.30 × 0.18 mm

### Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
$T_{min}$ = 0.987, $T_{max}$ = 1.000
12712 measured reflections
3528 independent reflections
2748 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.176
$S$ = 1.05
3528 reflections
168 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of the disordered benzene rings C1*A* –C6*A* and C1*B*–C6*B*, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2*B*···O1$^{i}$ | 0.88 | 2.12 | 2.9785 (15) | 166 |
| N3—H3*B*···O1$^{ii}$ | 0.88 | 2.08 | 2.9434 (14) | 168 |
| C10—H10*A*···O1$^{i}$ | 0.98 | 2.51 | 3.2384 (17) | 131 |
| C10—H10*B*···N1$^{iii}$ | 0.98 | 2.58 | 3.4566 (19) | 148 |
| C4*B*—H4*BA*···*Cg*1$^{iv}$ | 0.95 | 2.86 | 3.618 (5) | 138 |
| C4*A*—H4*AA*···*Cg*1$^{iv}$ | 0.95 | 2.76 | 3.590 (5) | 146 |
| C4*A*—H4*AA*···*Cg*2$^{iv}$ | 0.95 | 2.93 | 3.714 (5) | 141 |

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+3$; (ii) $-x+1, y, -z+\frac{7}{2}$; (iii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+2$; (iv) $x, -y+1, z-\frac{1}{2}$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

BN thanks the UGC for financial assistance through SAP and BSR one-time grants for the purchase of chemicals. HSY thanks the University of Mysore for research facilities. RJB wishes to acknowledge the NSF–MRI program (grant CHE-0619278) for funds to purchase the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5032).

## References

Beraldo, H. & Gambino, D. (2004). *Mini Rev. Med. Chem.* **4**, 31–39.
Beraldo, H., Sinisterra, R. D., Teixeira, L. R., Vieira, R. P. & Doretto, M. C. (2002). *Biochem. Biophys. Res. Commun.* **296**, 241–246.
Du, C. X., Guo, C., Hansell, E., Doyle, P. S., Caffrey, C. R., Holler, T. P., McKerrow, J. H. & Cohen, F. E. (2004). *J. Med. Chem.* **47**, 3212–3219.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kucukguzel, G., Kocatepe, A., De Clercq, E., Sahin, F. & Gulluce, M. (2006). *Eur. J. Med. Chem.* **41**, 353–359.
Naik, D. V. & Palenik, G. J. (1974). *Acta Cryst.* B**30**, 2396–2401.
Oxford Diffraction (2007). *CrysAlis PRO* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
Sarojini, B. K., Narayana, B., Bindya, S., Yathirajan, H. S. & Bolte, M. (2007). *Acta Cryst.* E**63**, o2946.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Teixeira, L. R., Sinisterra, R. D., Vieira, R. P., Doretto, M. C. & Beraldo, H. (2003). *J. Incl. Phenom. Macro. Chem.* **47**, 77–82.
Wang, J.-L., Jia, Y.-J. & Yu, M. (2004). *Acta Cryst.* E**60**, o662–o663.
Yathirajan, H. S., Bindya, S., Narayana, B., Sarojini, B. K. & Bolte, M. (2006). *Acta Cryst.* E**62**, o5925–o5926.

# supporting information

*Acta Cryst.* (2012). E**68**, o76–o77 [doi:10.1107/S160053681105255X]

## (2*E*)-2-[(3*E*)-4-Phenylbut-3-en-2-ylidene]hydrazinecarboxamide

**S. Samshuddin, Ray J. Butcher, Sema Ozturk Yıldırım, Mehmet Akkurt, B. Narayana and H. S. Yathirajan**

### S1. Comment

Semicarbazones presents a wide range of biological applications such as antitumoral, anticonvulsant, anti-trypanosomal, herbicidal and biocidal activities (Beraldo & Gambino, 2004; Beraldo *et al.*, 2002; Teixeira *et al.*, 2003). They can also be used as important intermediates in organic synthesis, mainly for obtaining heterocycle rings, such as thiazolidones, oxadiazoles, pyrazolidones, and thiadiazoles (Du *et al.*, 2004; Kucukguzel *et al.*, 2006)

Crystal structures of some semicarbazone derivatives, *viz*., acetone semicarbazone and benzaldehyde semicarbazone (Naik & Palenik, 1974); 3,4- methylenedioxybenzaldehyde semicarbazone (Wang *et al.*,2004); 4-(methylsulfanyl)benzaldehyde thiosemicarbazone (Yathirajan *et al.*, 2006) and 4-(Methylsulfanyl)benzaldehyde semicarbazone (Sarojini *et al.*, 2007) have been reported. In view of the importance of semicarbazones, the title compound (I) was prepared and its crystal structure is reported.

Fig. 1 shows the molecular structure of the title compound (I) with the disordered phenyl ring. The dihedral angle between the major and minor disorder components of the phenyl ring is 12.9 (2)°. The C7—C8—C9—C10, C7—C8—C9—N1, C10—C9—N1—N2, C8—C9—N1—N2, N1—N2—C11—N3 and N1—N2—C11—O1 torsion angles are -2.7 (2), 178.13 (13), -1.37 (19), 179.53 (10), -1.40 (17) and 179.22 (11)°, respectively, and indicate planarity in the molecule.

In the crystal, the molecules form centrosymmetric dimers with an $R_2^2(8)$ ring motif through a pair of N—H···O hydrogen bonds. These dimers are further connected into a three-dimensional network by intermolecular C—H···O and C—H···N hydrogen bonds (Table 1, Fig. 2). Weak intermolecular C—H···$\pi$ interactions further stabilize the crystal structure.

### S2. Experimental

To a mixture of a benzylidene acetone (1.46 g, 0.01 mol) and semicarbazide hydrochloride (1.12 g, 0.01 mol) in 50 ml ethanol was added a sodium acetate solution (2 g in 5 ml water) which was then refluxed for 4 h. The resultant solution was concentrated to half of its volume and poured into 50 ml ice-cold water. The precipitate thus formed was collected by filtration and purified by recrystallization from ethanol. The single crystal was grown from its absolute alcohol solution by slow evaporation. The yield was 74%. (M.pt. 455–459 K).

### S3. Refinement

The phenyl ring is disordered over two positions with refined site occupancies of 0.520 (17) and 0.480 (17). All H atoms were placed in idealised positions and refined in the riding model approximation [N—H = 0.88 Å, aromatic C—H = 0.95 Å and methyl C—H = 0.98 Å, and with $U_{iso}$(H) = 1.2 or 1.5 $U_{eq}$(parent atom)]. In the crystal structure, there is an 206 Å$^3$ void, but the low electron density (0.26 e.Å$^{-3}$) in the difference Fourier map suggests no solvent molecule occupying this void.

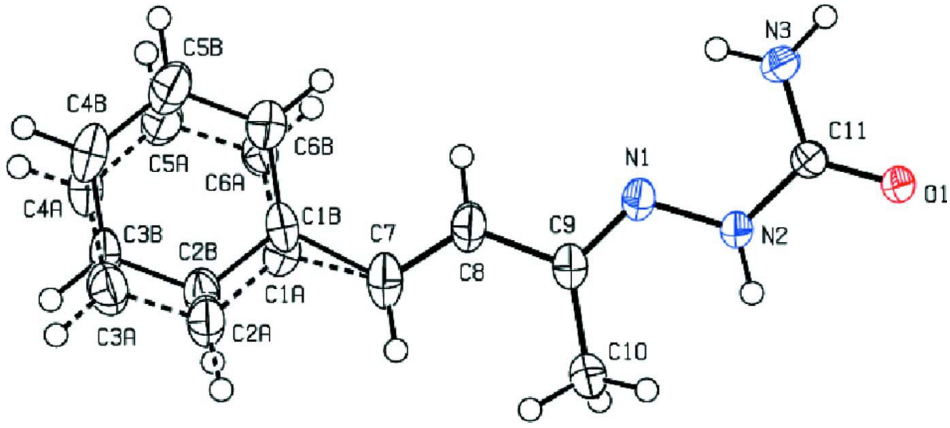



**Figure 1**

The disordered molecule (I) showing the atom labeling scheme. Atoms of the minor disorder components are joined with dashed lines. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

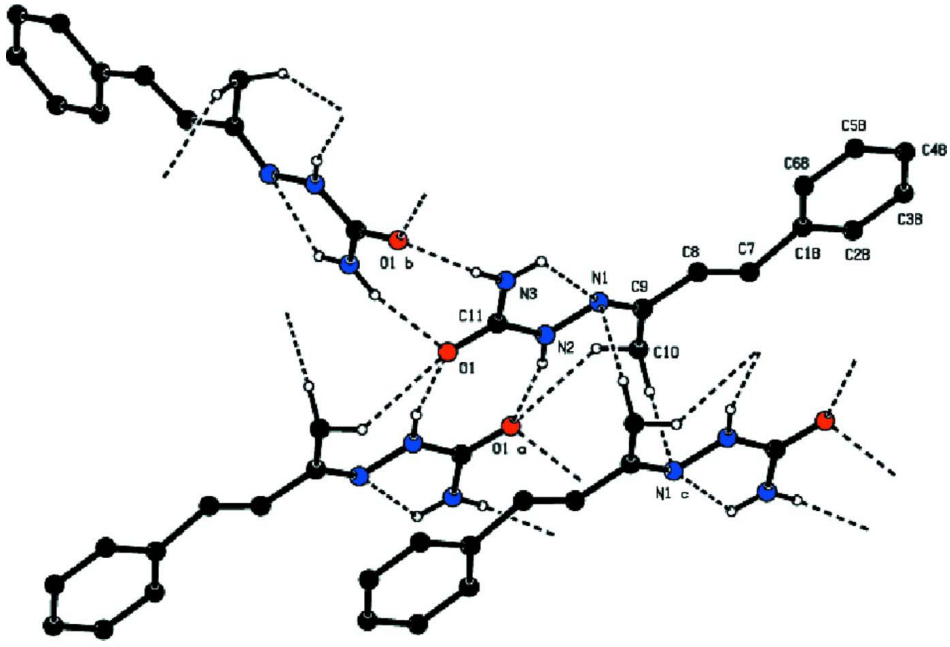



**Figure 2**

View of the N—H···O mediated dimers in (I) and their connections to other molecules by C—H···O and C—H···O hydrogen bonding.

### (2*E*)-2-[(3*E*)-4-Phenylbut-3-en-2-ylidene]hydrazinecarboxamide

*Crystal data*

$C_{11}H_{13}N_3O$
$M_r = 203.24$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 15.1094\ (8)$ Å
$b = 24.4445\ (11)$ Å
$c = 7.0368\ (4)$ Å
$\beta = 109.908\ (6)°$
$V = 2443.7\ (2)$ Å$^3$
$Z = 8$
$F(000) = 864$
$D_x = 1.105$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5469 reflections

$\theta$ = 3.0–30.9°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 123 K

Prism, colourless
0.40 × 0.30 × 0.18 mm

*Data collection*

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
$T_{min}$ = 0.987, $T_{max}$ = 1.000

12712 measured reflections
3528 independent reflections
2748 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.026
$\theta_{max}$ = 30.9°, $\theta_{min}$ = 3.0°
$h$ = −20→20
$k$ = −34→26
$l$ = −7→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.176
$S$ = 1.05
3528 reflections
168 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0981P)^2 + 0.6768P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted $R$-factors $wR$ and all goodnesses of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -$R$-factor-obs *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$-factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.36888 (6) | 0.23438 (4) | 1.62948 (12) | 0.0323 (3) | |
| N1 | 0.34428 (7) | 0.30220 (5) | 1.17621 (15) | 0.0337 (3) | |
| N2 | 0.31934 (7) | 0.27704 (4) | 1.32596 (14) | 0.0310 (3) | |
| N3 | 0.47771 (7) | 0.26174 (6) | 1.49376 (16) | 0.0458 (4) | |
| C1B | 0.3063 (5) | 0.40506 (19) | 0.5724 (7) | 0.0301 (9) | 0.520 (17) |
| C2B | 0.2428 (5) | 0.4316 (2) | 0.4074 (6) | 0.0354 (10) | 0.520 (17) |
| C3B | 0.2747 (6) | 0.45824 (19) | 0.2685 (6) | 0.0405 (13) | 0.520 (17) |
| C4B | 0.3701 (6) | 0.45829 (14) | 0.2947 (8) | 0.0409 (13) | 0.520 (17) |
| C5B | 0.4336 (6) | 0.4317 (2) | 0.4598 (11) | 0.0469 (14) | 0.520 (17) |
| C6B | 0.4017 (5) | 0.4051 (2) | 0.5986 (11) | 0.0434 (11) | 0.520 (17) |
| C7 | 0.25908 (11) | 0.37670 (5) | 0.71144 (19) | 0.0393 (4) | |
| C8 | 0.31231 (10) | 0.35103 (5) | 0.87866 (19) | 0.0375 (4) | |
| C9 | 0.27918 (9) | 0.32596 (5) | 1.03065 (18) | 0.0327 (3) | |

| | | | | | |
|---|---|---|---|---|---|
| C10 | 0.17796 (10) | 0.32953 (6) | 1.0129 (2) | 0.0395 (4) | |
| C11 | 0.38883 (8) | 0.25686 (5) | 1.49014 (17) | 0.0304 (3) | |
| C3A | 0.2443 (5) | 0.4630 (2) | 0.2639 (7) | 0.0458 (13) | 0.480 (17) |
| C4A | 0.3345 (6) | 0.45553 (15) | 0.2576 (7) | 0.0378 (13) | 0.480 (17) |
| C5A | 0.3972 (6) | 0.4208 (2) | 0.3956 (10) | 0.0408 (14) | 0.480 (17) |
| C6A | 0.3697 (5) | 0.39363 (19) | 0.5398 (9) | 0.0337 (11) | 0.480 (17) |
| C2A | 0.2169 (4) | 0.4358 (2) | 0.4081 (7) | 0.0402 (11) | 0.480 (17) |
| C1A | 0.2795 (4) | 0.40110 (19) | 0.5461 (6) | 0.0276 (10) | 0.480 (17) |
| H7A | 0.19470 | 0.37930 | 0.69980 | 0.0470* | |
| H3BA | 0.23130 | 0.47640 | 0.15570 | 0.0490* | 0.520 (17) |
| H5BA | 0.49890 | 0.43180 | 0.47770 | 0.0560* | 0.520 (17) |
| H6BA | 0.44510 | 0.38700 | 0.71150 | 0.0520* | 0.520 (17) |
| H10A | 0.17330 | 0.33360 | 1.14770 | 0.0590* | |
| H10B | 0.14520 | 0.29610 | 0.94940 | 0.0590* | |
| H10C | 0.14890 | 0.36120 | 0.92980 | 0.0590* | |
| H4BA | 0.39200 | 0.47650 | 0.19980 | 0.0490* | 0.520 (17) |
| H8A | 0.37790 | 0.34890 | 0.90100 | 0.0450* | |
| H2B | 0.25980 | 0.27410 | 1.31570 | 0.0370* | |
| H3B | 0.52440 | 0.24890 | 1.59690 | 0.0550* | |
| H3C | 0.48940 | 0.27780 | 1.39300 | 0.0550* | |
| H2BA | 0.17760 | 0.43160 | 0.38950 | 0.0420* | 0.520 (17) |
| H2AA | 0.15520 | 0.44090 | 0.41240 | 0.0480* | 0.480 (17) |
| H3AA | 0.20150 | 0.48670 | 0.16960 | 0.0550* | 0.480 (17) |
| H4AA | 0.35330 | 0.47410 | 0.15910 | 0.0450* | 0.480 (17) |
| H5AA | 0.45880 | 0.41570 | 0.39130 | 0.0490* | 0.480 (17) |
| H6AA | 0.41250 | 0.36990 | 0.63410 | 0.0400* | 0.480 (17) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0234 (4) | 0.0505 (6) | 0.0224 (4) | 0.0004 (3) | 0.0069 (3) | 0.0096 (3) |
| N1 | 0.0334 (5) | 0.0409 (6) | 0.0276 (5) | −0.0035 (4) | 0.0113 (4) | 0.0100 (4) |
| N2 | 0.0239 (4) | 0.0438 (6) | 0.0243 (5) | −0.0019 (4) | 0.0069 (4) | 0.0107 (4) |
| N3 | 0.0224 (5) | 0.0857 (10) | 0.0292 (5) | −0.0024 (5) | 0.0086 (4) | 0.0155 (5) |
| C1B | 0.045 (2) | 0.0227 (15) | 0.0208 (13) | 0.0012 (14) | 0.0088 (15) | 0.0032 (10) |
| C2B | 0.045 (2) | 0.0379 (17) | 0.0259 (14) | 0.0056 (16) | 0.0156 (14) | 0.0043 (11) |
| C3B | 0.059 (3) | 0.0386 (18) | 0.0276 (14) | 0.0065 (18) | 0.0197 (17) | 0.0131 (11) |
| C4B | 0.065 (3) | 0.0320 (15) | 0.0343 (18) | −0.0086 (16) | 0.028 (2) | 0.0002 (13) |
| C5B | 0.055 (3) | 0.0457 (19) | 0.050 (2) | −0.0018 (19) | 0.031 (2) | 0.0083 (17) |
| C6B | 0.046 (2) | 0.0458 (19) | 0.041 (2) | 0.0010 (18) | 0.0182 (19) | 0.0113 (17) |
| C7 | 0.0638 (9) | 0.0303 (6) | 0.0298 (6) | 0.0083 (6) | 0.0238 (6) | 0.0061 (5) |
| C8 | 0.0488 (7) | 0.0349 (6) | 0.0335 (6) | 0.0037 (5) | 0.0202 (6) | 0.0097 (5) |
| C9 | 0.0389 (6) | 0.0323 (6) | 0.0279 (5) | 0.0005 (5) | 0.0126 (5) | 0.0065 (4) |
| C10 | 0.0397 (7) | 0.0478 (8) | 0.0320 (6) | 0.0081 (5) | 0.0135 (5) | 0.0127 (5) |
| C11 | 0.0240 (5) | 0.0424 (7) | 0.0238 (5) | −0.0017 (4) | 0.0067 (4) | 0.0040 (4) |
| C3A | 0.056 (3) | 0.044 (2) | 0.0397 (18) | 0.0139 (18) | 0.0194 (18) | 0.0134 (14) |
| C4A | 0.055 (3) | 0.0314 (17) | 0.0291 (15) | −0.0016 (18) | 0.017 (2) | 0.0062 (12) |
| C5A | 0.045 (3) | 0.0402 (19) | 0.044 (2) | 0.0033 (16) | 0.024 (2) | 0.0081 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C6A | 0.038 (2) | 0.0309 (16) | 0.0338 (19) | 0.0064 (14) | 0.0144 (18) | 0.0112 (13) |
| C2A | 0.053 (2) | 0.0369 (19) | 0.0353 (17) | 0.0087 (17) | 0.0212 (16) | 0.0099 (13) |
| C1A | 0.038 (2) | 0.0219 (14) | 0.0268 (15) | −0.0032 (14) | 0.0161 (14) | −0.0043 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C11 | 1.2472 (15) | C5A—C6A | 1.389 (10) |
| N1—N2 | 1.3788 (15) | C5B—C6B | 1.389 (10) |
| N1—C9 | 1.2915 (17) | C7—C8 | 1.3344 (18) |
| N2—C11 | 1.3618 (15) | C8—C9 | 1.4610 (19) |
| N3—C11 | 1.3398 (17) | C9—C10 | 1.494 (2) |
| N2—H2B | 0.8800 | C2A—H2AA | 0.9500 |
| N3—H3B | 0.8800 | C2B—H2BA | 0.9500 |
| N3—H3C | 0.8800 | C3A—H3AA | 0.9500 |
| C1A—C7 | 1.432 (5) | C3B—H3BA | 0.9500 |
| C1A—C6A | 1.391 (10) | C4A—H4AA | 0.9500 |
| C1A—C2A | 1.390 (7) | C4B—H4BA | 0.9500 |
| C1B—C2B | 1.390 (7) | C5A—H5AA | 0.9500 |
| C1B—C6B | 1.390 (11) | C5B—H5BA | 0.9500 |
| C1B—C7 | 1.556 (6) | C6A—H6AA | 0.9500 |
| C2A—C3A | 1.389 (8) | C6B—H6BA | 0.9500 |
| C2B—C3B | 1.390 (9) | C7—H7A | 0.9500 |
| C3A—C4A | 1.391 (12) | C8—H8A | 0.9500 |
| C3B—C4B | 1.390 (13) | C10—H10B | 0.9800 |
| C4A—C5A | 1.391 (9) | C10—H10C | 0.9800 |
| C4B—C5B | 1.391 (9) | C10—H10A | 0.9800 |
| | | | |
| N2—N1—C9 | 118.31 (11) | C3A—C2A—H2AA | 120.00 |
| N1—N2—C11 | 118.53 (11) | C1A—C2A—H2AA | 120.00 |
| N1—N2—H2B | 121.00 | C3B—C2B—H2BA | 120.00 |
| C11—N2—H2B | 121.00 | C1B—C2B—H2BA | 120.00 |
| C11—N3—H3B | 120.00 | C4A—C3A—H3AA | 120.00 |
| H3B—N3—H3C | 120.00 | C2A—C3A—H3AA | 120.00 |
| C11—N3—H3C | 120.00 | C4B—C3B—H3BA | 120.00 |
| C6A—C1A—C7 | 117.1 (4) | C2B—C3B—H3BA | 120.00 |
| C2A—C1A—C7 | 122.6 (5) | C5A—C4A—H4AA | 120.00 |
| C2A—C1A—C6A | 119.9 (5) | C3A—C4A—H4AA | 120.00 |
| C2B—C1B—C7 | 113.5 (6) | C5B—C4B—H4BA | 120.00 |
| C2B—C1B—C6B | 120.0 (6) | C3B—C4B—H4BA | 120.00 |
| C6B—C1B—C7 | 126.4 (5) | C4A—C5A—H5AA | 120.00 |
| C1A—C2A—C3A | 120.1 (6) | C6A—C5A—H5AA | 120.00 |
| C1B—C2B—C3B | 120.0 (7) | C4B—C5B—H5BA | 120.00 |
| C2A—C3A—C4A | 120.0 (5) | C6B—C5B—H5BA | 120.00 |
| C2B—C3B—C4B | 120.0 (5) | C5A—C6A—H6AA | 120.00 |
| C3A—C4A—C5A | 120.0 (6) | C1A—C6A—H6AA | 120.00 |
| C3B—C4B—C5B | 120.0 (6) | C5B—C6B—H6BA | 120.00 |
| C4A—C5A—C6A | 120.0 (8) | C1B—C6B—H6BA | 120.00 |
| C4B—C5B—C6B | 120.0 (8) | C1A—C7—H7A | 114.00 |

| | | | |
|---|---|---|---|
| C1A—C6A—C5A | 120.0 (6) | C1B—C7—H7A | 126.00 |
| C1B—C6B—C5B | 120.0 (6) | C8—C7—H7A | 114.00 |
| C1B—C7—C8 | 119.7 (3) | C9—C8—H8A | 117.00 |
| C1A—C7—C8 | 132.9 (3) | C7—C8—H8A | 117.00 |
| C7—C8—C9 | 126.08 (15) | C9—C10—H10C | 110.00 |
| N1—C9—C8 | 114.27 (13) | C9—C10—H10B | 109.00 |
| C8—C9—C10 | 120.69 (11) | H10B—C10—H10C | 109.00 |
| N1—C9—C10 | 125.04 (12) | H10A—C10—H10B | 110.00 |
| N2—C11—N3 | 117.53 (11) | H10A—C10—H10C | 109.00 |
| O1—C11—N3 | 122.23 (11) | C9—C10—H10A | 110.00 |
| O1—C11—N2 | 120.24 (12) | | |

| | | | |
|---|---|---|---|
| C9—N1—N2—C11 | −173.34 (11) | C2B—C1B—C7—C8 | −178.1 (3) |
| N2—N1—C9—C8 | −179.53 (10) | C6B—C1B—C7—C8 | 2.8 (6) |
| N2—N1—C9—C10 | 1.37 (19) | C1B—C2B—C3B—C4B | 0.0 (7) |
| N1—N2—C11—O1 | 179.22 (11) | C2B—C3B—C4B—C5B | 0.0 (7) |
| N1—N2—C11—N3 | −1.40 (17) | C3B—C4B—C5B—C6B | 0.0 (8) |
| C6B—C1B—C2B—C3B | 0.0 (7) | C4B—C5B—C6B—C1B | 0.0 (8) |
| C7—C1B—C2B—C3B | −179.2 (4) | C1B—C7—C8—C9 | 175.5 (2) |
| C2B—C1B—C6B—C5B | 0.0 (8) | C7—C8—C9—N1 | 178.13 (13) |
| C7—C1B—C6B—C5B | 179.1 (4) | C7—C8—C9—C10 | −2.7 (2) |

*Hydrogen-bond geometry (Å, º)*

Cg1 and Cg2 are the centroids of the disordered benzene rings C1A –C6A and C1B–C6B, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*B*···O1$^{i}$ | 0.88 | 2.12 | 2.9785 (15) | 166 |
| N3—H3*B*···O1$^{ii}$ | 0.88 | 2.08 | 2.9434 (14) | 168 |
| N3—H3*C*···N1 | 0.88 | 2.28 | 2.6397 (16) | 104 |
| C10—H10*A*···O1$^{i}$ | 0.98 | 2.51 | 3.2384 (17) | 131 |
| C10—H10*B*···N1$^{iii}$ | 0.98 | 2.58 | 3.4566 (19) | 148 |
| C4*B*—H4*BA*···*Cg*1$^{iv}$ | 0.95 | 2.86 | 3.618 (5) | 138 |
| C4*A*—H4*AA*···*Cg*1$^{iv}$ | 0.95 | 2.76 | 3.590 (5) | 146 |
| C4*A*—H4*AA*···*Cg*2$^{iv}$ | 0.95 | 2.93 | 3.714 (5) | 141 |

Symmetry codes: (i) −*x*+1/2, −*y*+1/2, −*z*+3; (ii) −*x*+1, *y*, −*z*+7/2; (iii) −*x*+1/2, −*y*+1/2, −*z*+2; (iv) *x*, −*y*+1, *z*−1/2.